

Kraft lignins – Dry matter content – Oven-drying method

0 Introduction

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1 Scope

This Innventia Test Method document describes a method for the determination of the dry matter content of lignins, that is, the residue after drying at specified conditions. Note that any matter that is volatile at the drying conditions is not included in the dry matter content.

The method is mainly intended for the investigation of kraft lignins, but may, however, also be used with other lignins, for instance soda lignins, lignosulphonates or organosolv lignins.

2 Normative references

No referenced documents are indispensable for the application of this document.

3 Terms and definitions

For the purposes of this Innventia Test Method, the following definitions apply:

3.1 Kraft lignin

Solid matter containing mainly degraded lignin isolated from a kraft pulping process (eg. Isolated from a kraft black liquor).

3.2 Dry matter content

The ratio of the mass of a sample after drying at a temperature of $105\text{ °C} \pm 2\text{ °C}$ during 2 to 20 h to the mass before drying. The dry matter content is expressed in weight-%.

4 Principle

The sample is dried at a temperature of $105\text{ °C} \pm 2\text{ °C}$. The mass before and after the drying is determined gravimetrically. The dry matter content of sample is calculated as the ratio of mass after to the mass before the drying.

5 Equipment and reagents

Ordinary laboratory equipment and the following:

5.1 *Aluminium pans.*

5.2 *Analytical balance*, accurate to 1 mg.

5.3 *Drying oven*, ($105\text{ °C} \pm 2\text{ °C}$)

5.4 *Desiccator*, containing desiccant.

6 Sampling

The sampling procedure is not covered by this method. Make sure that the test portions taken are representative of the samples received.

7 Procedure

Handle the samples and equipment with clean, dry, gloves.

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- 7.1 Samples are analysed in duplicate
- 7.2 Weigh the empty aluminium pan to the nearest 1 mg.
- 7.3 Add $1 \text{ g} \pm 0.5 \text{ g}$ sample to the pan and record the weight of the sample plus weighing pan to the nearest 1 mg
- 7.4 Place the sample in the oven and dry at $105 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ for not less than 2 hours and not more than 20 hours.
- 7.5 Remove the sample from the oven and allow it to cool at room temperature in a desiccator. Weigh the pan containing the oven-dried sample to the nearest 1 mg and record the weight.

8 Calculation

Calculate the dry matter content, w_{dm} , of the kraft lignin sample expressed as weight-% from the expression:

$$w_{dm} = \frac{m_1}{m_0} \times 100 \quad (1)$$

where:

- w_{dm} is the dry matter content expressed as a percentage mass fraction (weight-%);
 m_1 is the mass in grams of the oven dried sample;
 m_0 is the mass in grams of the sample before drying;

For duplicate samples, the mean w_{dm} of the samples is calculated.

Report the results to the first decimal place.

9 Report

The test report shall include the following information:

- a) A reference to this Innventia Test Method;
- b) Date and place of testing;
- c) Identification of the sample tested;
- d) The results expressed as the dry matter content in weight-%;
- e) Information regarding any departure from the procedure described in this Innventia Test Method and/or any other

circumstances that may have affected the result.

10 Precision

10.1 Repeatability

The dry matter content of a lignin sample isolated from softwood kraft black liquor was determined using the procedure described (ie. average of duplicate determinations). The mean value of three parallel determinations during repeatability conditions are given in Table 1.

Table 1. Dry matter content in lignin isolated from softwood kraft black liquor, in ($n = 3$).

Sample	Mean value	Repeatability standard deviation*
Softwood kraft lignin	96.6 %	0.1 %

* The standard deviation with two decimal points was 0.05 %.

10.2 Reproducibility

Reproducibility information is currently not available for this test method.

11 References

ISO 638:2008 Paper, board and pulps – Determination of dry matter content – Oven-drying method

NREL/TP-510-42621 (2008) Determination of Total Solids in Biomass and Total Dissolved Solids in Liquid Process Samples

Biorefinery Test Methods are issued and recommended by Innventia for the pulp, paper and biorefinery industries.

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